

One of the significant disadvantages of quartz glass-based materials is their tendency to crystallize cristobalite during firing and, as a consequence, a significant deterioration in performance. In order to prevent crystallization of quartz ceramics during sintering, a number of additives are used. However, all known options have some disadvantages, namely, relatively low strength values and increased coefficient of linear thermal expansion of products.

In this regard, a promising area of research is to study the effect of aluminum phosphate additives on the properties of quartz ceramics. According to the totality of properties, the addition of $AlPO_4$ in an amount of 20 wt % has the most positive effect on the characteristics of quartz ceramics. According to differential thermal analysis, it is noted that up to a temperature of 1,200 °C, no noticeable phase and modification transformations occur in the base mixtures. In the course of the work, it was found that the most appropriate firing mode is as follows: oxidizing medium; the products are immersed in a hot furnace, after being held at the maximum temperature, they are removed from the hot furnace, cooling occurs slowly in the air. The materials obtained in this way are characterized by the following properties: $\alpha = 31.6 \cdot 10^{-7} \text{ deg}^{-1}$; $\sigma_{comp} = 153 \text{ MPa}$; $P = 2.7 \%$; $tg\delta$ and ϵ (frequency 10^{10} Hz , temperature 20 °C) 0.001 and 10, respectively. It is noted that the main crystal phase prevailing in the samples is aluminum phosphate. The microstructure of the material is characterized by the presence of a small number of residual pores and a densely sintered shard.

The proposed solution will significantly reduce power consumption, improve production performance and increase the basic properties of polyfunctional quartz ceramic products

Keywords: quartz ceramics, aluminum phosphate, cristobalite, crystallization, slip casting, firing mode

DETERMINATION OF THE INFLUENCE OF ALUMINUM PHOSPHATE ON THE PROPERTIES OF QUARTZ CERAMICS

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1. Introduction

The properties of ceramic materials are determined by their chemical, phase, mineralogical composition, as well as crystal lattice structure, density and porosity of the fired product. The desired properties of products can only be realized by the correct choice of material composition. In addition, compliance with a strict manufacturing technology provides the required characteristics: density, porosity, crystal size distribution, etc. [1, 2].

A promising direction in the production of ceramic materials is the possibility of regulating their basic physical and chemical properties. Materials based on quartz glass are of great scientific and practical interest [3–6].

Quartz glass is a single-component silicate glass made up of virtually only silica (SiO_2). Quartz glass has a number of valuable physical and chemical properties: heat resistance, fire resistance, chemical and radiation resistance, transparency in a wide wavelength range, including radio frequencies, optical and acoustic homogeneity, high electric insulation properties, etc. [7–10].

Quartz ceramics is a conventionally common name for products obtained by ceramic technology from silica. The starting material for the production of quartz ceramics is powders of various types of quartz glass. Thus, the basis of

quartz ceramics is not a crystal, but an amorphous vitreous phase. In the process of heat treatment of quartz glass products, there are prerequisites for the formation and growth of undesirable crystal phases, mainly cristobalite. As a result, the performance of quartz ceramic products is significantly degraded. Therefore, solving this problem is a very urgent and necessary task.

2. Literature review and problem statement

The work [11] presents the results of studies of the crystallization ability of quartz glass. It is shown that cristobalite is a product of spontaneous crystallization of quartz glass, which occurs during production or, more often, operation at temperatures above 1,000–1,100 °C. The results indicate an analogy between the structures of α -cristobalite and quartz glass.

High-temperature cristobalite crystallizing in a cubic system has the most “regular” structure, geometrically similar to that of diamond or metallic silicon. The ideal cristobalite structure is of the three-layer type, but observed only at high temperatures (above 1,500 °C) for very pure cristobalite. At low temperatures, two-layer motifs appear in the cristobalite structure, especially in the presence of ion

impurities. This brings the structure of such defective cristobalite closer to that of tridymite. α -cristobalite is a brittle white material that has a tetragonal crystal lattice.

The transformation of quartz into cristobalite comes from the surface of the grains. For large grains (2–3 mm), the transformation also proceeds through the cracks that are formed during heat treatment. Usually, the front of the formation of a new phase (cristobalite) exactly copies the shape of the quartz grain. It is known that these transformations occur in the solid phase [12–14]. It was found in [12] that the process of quartz transformation into cristobalite, which was considered direct, is a sequential reaction with the appearance of an intermediate transition phase. It was found that the rate of the transformation reaction depends significantly on the type of starting material. Ex situ observations [13] showed that the formation of cristobalite from quartz demonstrates a noticeable dependence on the interplanar spacings of the crystal lattice of the initial quartz. Cristobalite formation is markedly enhanced at temperatures above 1200 °C.

It is noted [14] that the addition of 2 % alkali metal oxide (Na_2O , Li_2O , K_2O) to quartz when heated in an oxidizing medium leads to the appearance of cristobalite as an intermediate phase at temperatures below 1,000 °C. The process of cristobalitization is intensified by numerous foreign ions, the specific action of alkali cations being unrelated to the formation of a liquid phase by them or the amount thereof, although significantly activating this process.

When cooling a partially crystallized product below 1,000 °C, due to the difference in the thermal expansion coefficient, stresses begin to appear in it, stretching in the cristobalite layers and compressing in the glass. These stresses become especially high after the $\beta \rightarrow \alpha$ transition (at ~230 °C) [15] when the cristobalite volume abruptly decreases by about 4 %. At the same time, the cristobalite layer and the adjacent glass areas crack and the product, even if it retains its integrity, loses transparency and strength, becoming unsuitable for further use.

Crystallization of quartz glass is strongly influenced by various impurities [16]. Obviously, uneven distribution of the impurity in the glass sample should have a strong effect on the change in crystallization rate over time.

Crystal growth from crystallization nuclei begins from the surface. Surface contamination of quartz glass can affect both the growth rate of crystals and the number of nuclei. Even a touch of fingers to quartz glass causes a slight deposition of mineral salts. The crystallization ability of amorphous silica is determined by the purity of the starting material (presence of impurities), presence of water, gas medium during heat treatment, a number of technological factors, etc. [17].

An option to overcome these difficulties can be the introduction of additives into the ceramic slip. This approach was used in [18–21]. In order to prevent crystallization of quartz ceramics during sintering, boron oxide [18], anoxic boron-containing sintering activators [18, 19], kaolin [20], nanodispersed silica [21], etc. were studied.

However, all the proposed options have a number of disadvantages, namely, relatively low strength values and increased coefficient of linear thermal expansion of products. This indicates the presence of the cristobalite phase in the finished product. It is known that the magnitude of the thermal expansion of crystalline substances, in particular, pure

oxides [22], is closely related to the structure of the crystal lattice and the forces of interionic bonds therein.

The simpler the crystal structure of the compound and the lower the bond strength between the ions in it, the greater the absolute value of thermal expansion.

In addition, when using a boron-containing additive, the technology of obtaining products very often involves several steps of firing due to its oxidation and uneven distribution in the material [18]. Multiple firing complicates production technology and increases power consumption.

Thus, the issue of finding an effective additive to control the properties of quartz ceramics and reduce the growth of the cristobalite phase in the finished product, as well as during its operation, remains unresolved.

All this suggests it expedient to conduct a study on the effect of adding aluminum phosphate in the production of quartz ceramics on its structure and properties.

It is known that aluminum phosphate (AlPO_4) has a structure similar to quartz glass [23], so it is easily embedded in the SiO_2 lattice. Uniform distribution of aluminum phosphate particles in quartz glass is assumed to compact the structure of ceramic materials.

3. The aim and objectives of the study

The aim of the study is to determine the effect of aluminum phosphate on the properties of quartz ceramics.

To achieve the aim, the following objectives were set:

- to determine the factors that significantly affect the properties of quartz ceramics;
- to choose the optimal mode of firing quartz ceramic samples with reduced porosity;
- to determine the crystal-phase composition of materials with the best values of the studied properties.

4. Materials and methods for studying the effect of aluminum phosphate on the properties of quartz ceramics

Broken transparent quartz glass was used as a starting material for quartz ceramics. Initially, it was washed with 10 % hydrochloric acid, then pre-ground in a porcelain drum without adding water before passing through a sieve No. 0365.

Aluminum phosphate was used as a chemically pure $\text{Al}(\text{H}_2\text{PO}_4)_3$ reagent. Aluminum phosphate was pretreated at a temperature of 1,300 °C for 1 hour until complete removal of $(\text{OH})^1$ groups. The presence of a hydroxyl group during subsequent firing will lead to the undesirable pore formation and deterioration in the performance of products.

Aluminum phosphate was added to the base mixtures in an amount of 10, 15 and 20 wt %. Samples for determining the properties were prepared by slip casting into 20 % moisture plaster molds. The firing was carried out in an electric furnace in a temperature range of 1,100–1,250 °C.

The open porosity (P), water absorption (W) and apparent density (d) were evaluated by standard hydrostatic weighing.

The ultimate compressive strength (σ_{comp}) was determined by the maximum load that the samples withstand without destruction on a PSU-10 hydraulic press.

The coefficient of linear thermal expansion (CLTE, α) was determined by a DKV-5A automatic quartz dilatometer.

The measurement of the dielectric constant (ϵ) and dielectric loss tangent ($\text{tg}\delta$) was carried out on a measuring device consisting of a G4-83 generator, S4-11 spectrum analyzer and biconical cavity. The latter was connected according to the per-pass scheme. The measurements were made at 10^{10} Hz at 20°C .

The nature of thermal transformations of raw materials and composite mixtures was studied by differential thermal analysis using a derivatograph of the F. Paulik, J. Paulik, L. Erdey Q-1000 (Hungary) system, heating rate $5^\circ\text{C}/\text{min}$.

The crystal phase composition of the developed materials was studied by X-ray phase analysis (XPA) using a DRON-3 diffractometer (Russia) (Co-K α radiation).

To study the microstructure of the fired sample, a "REM - 106 - I" scanning electron microscope (Russia) was used.

5. Results of studies on the effect of aluminum phosphate on the properties of quartz ceramics

5.1. Determination of factors that significantly affect the properties of quartz ceramics

The technology of quartz ceramics has a number of problems: no shrinkage of the semi-finished product with the slip method of production; high porosity of products after firing and, as a result, low strength. The growth of the undesirable crystal phase (cristobalite) during the heat treatment of products leads to a deterioration many performance characteristics.

These drawbacks can be eliminated by adjusting the chemical composition of the base mixture, so aluminum phosphate in amounts of 10, 15 and 20 wt % was added to broken quartz glass. When making samples by slip casting, it was found that a slip with aluminum phosphate is much more technologically advanced than a slip made of pure quartz glass [24]. Samples are cast easier, without voids, harden faster, easier to remove from the mold, stronger, the mold does not need special pre-treatment.

The samples were fired in an oxidizing medium at $1,250^\circ\text{C}$ for 1 hour. The values of the properties of the materials under study are presented in Table 1.

The most preferred values of properties after firing are characteristic of ceramics of composition No. 3. The nature of thermal transformations in the initial mixture of the specified composition was assessed using differential thermal analysis (Fig. 1).

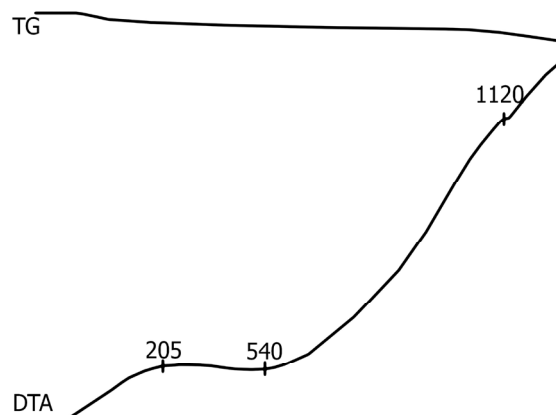


Fig. 1. Differential thermal analysis of the base mixture of composition No. 3

It was found that up to a temperature of $1,200^\circ\text{C}$, no noticeable phase and modification transformations occur in the samples, which should indicate the stability of the structure under study.

5.2. Selection of the optimal firing mode for quartz ceramics

To determine the effect of the firing mode and conditions (Table 2) on the properties and quality of the studied ceramic materials, composition No. 3 was chosen as the base.

The effect of firing mode on the properties of the materials obtained is shown in Fig. 2, 3.

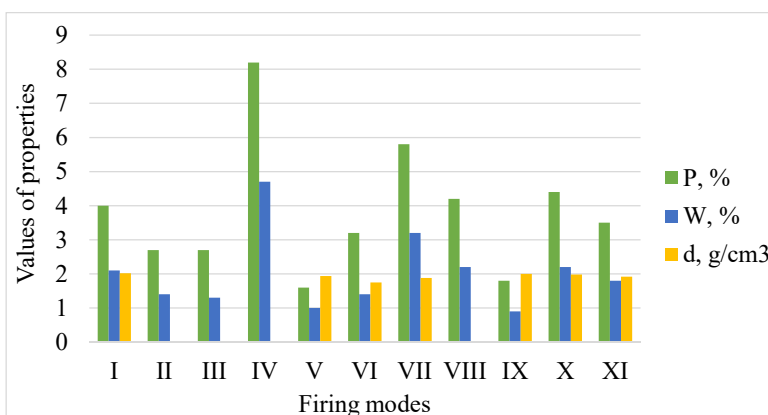


Fig. 2. Effect of firing mode on porosity, water absorption and density of samples

Table 1
Main physical and chemical properties of the studied samples

Com-position No.	Chemical composition, wt %		Sample properties		
	SiO ₂	Al(PO ₄) ₃	P, %	W, %	σ_{comp} , MPa
0	100	0	19.1	11.2	63
1	90	10	18.8	10.6	65
2	85	15	8.2	4.7	65
3	80	20	5.1	2.7	101

Thus, it was found that minimum porosity values are characteristic of samples fired according to modes V, IX, II, III, corresponding to an increase in its values. The firing temperature is $1,100$ – $1,200^\circ\text{C}$, the firing time is 1–2 h, the firing medium is oxidizing. The samples fired in the sand backfill (mode IX) were quite brittle, so the ultimate compressive strength and CLTE could not be measured. In this case, the values of the ultimate compressive strength from maximum to minimum, depending on the firing mode, are arranged as follows: II, VIII, XI, VI. According to the totality of the obtained properties ($\alpha=31.6 \cdot 10^{-7} \text{ deg}^{-1}$; $\sigma_{\text{comp}}=153 \text{ MPa}$; $P=2.7\%$), firing mode II is most preferable.

Table 2

Firing modes and conditions for ceramic materials

Firing mode	Maximum firing temperature, °C	Holding time at maximum temperature, h	Features of firing mode and conditions
I	1,100	1	oxidizing medium, the samples were immersed in a hot furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air
II	1,100	2	oxidizing medium, the samples were immersed in a hot furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air
III	1,200	2	oxidizing medium, the samples were immersed in a hot furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air
IV	1,250	1	oxidizing medium, the samples were immersed in a cold furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air
V	1,250	1	oxidizing medium, the samples were immersed in a hot furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air
VI	1,100	2	firing the samples in the sand, the samples were immersed in a hot furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air
VII	1,100	2	firing the samples in alumina, the samples were immersed in a hot furnace, after being held at the maximum temperature they were removed from the hot furnace; the samples were cooled in the air
VIII	1,100	2	firing the samples in a chromium oxide backfill, the samples were immersed in a hot furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air
IX	1,200	2	firing the samples in the sand, the samples were immersed in a hot furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air
X	1,200	2	firing the samples in alumina, the samples were immersed in a hot furnace, after being held at the maximum temperature they were removed from the hot furnace; the samples were cooled in the air
XI	1,200	2	firing the samples in a chromium oxide backfill, specimens were immersed in a hot furnace, after being held at the maximum temperature, they were removed from the hot furnace; the samples were cooled in the air

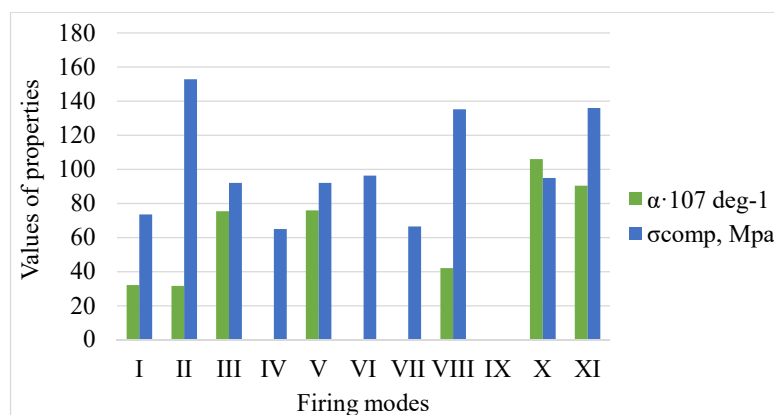


Fig. 3. Effect of firing mode on ultimate compressive strength of samples

For the ceramics obtained by the above method, the dielectric constant ϵ and the dielectric loss tangent $\text{tg}\delta$ were determined, which at a frequency of 10^{10} Hz and at a temperature of 20 °C are 10 and 0.001, respectively.

5. 3. Determination of crystal-phase composition of materials with the best values of the studied properties

The crystal-phase composition of the developed materials is shown in Fig. 4, 5.

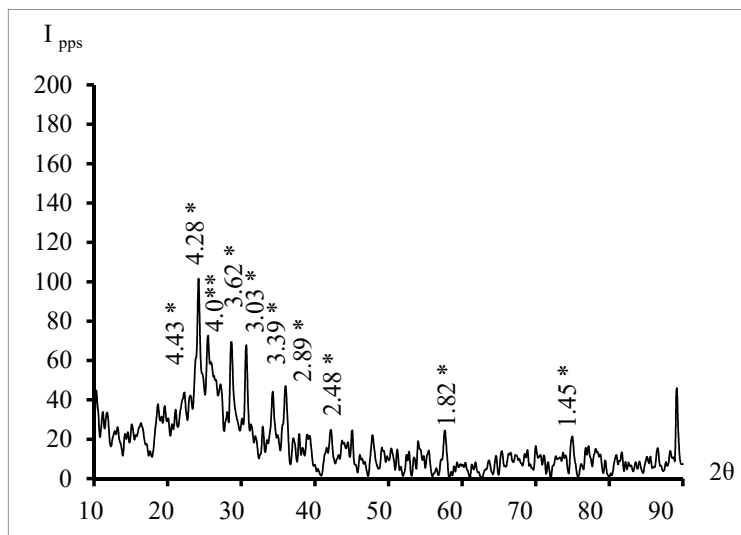


Fig. 4. Diffraction pattern of ceramic material of composition No. 3 before firing: * – AlPO_4 ; ** – SiO_2

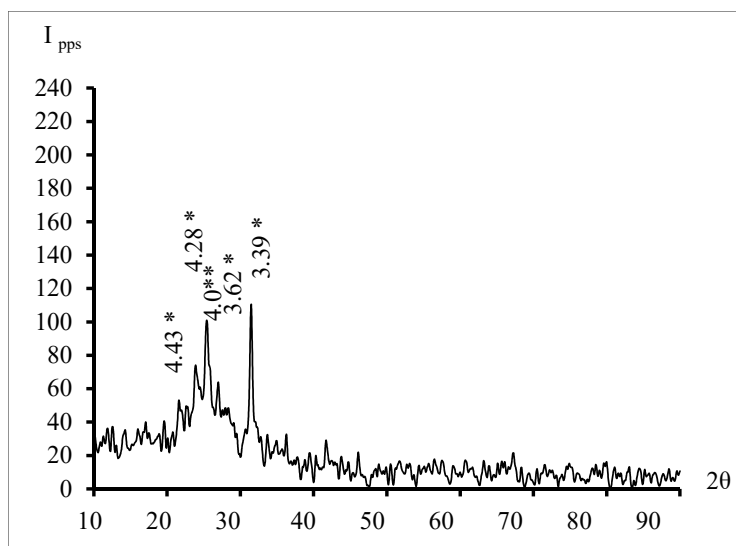


Fig. 5. Diffraction pattern of ceramic material of composition No. 3, firing mode II: * – AlPO_4 ; ** – SiO_2

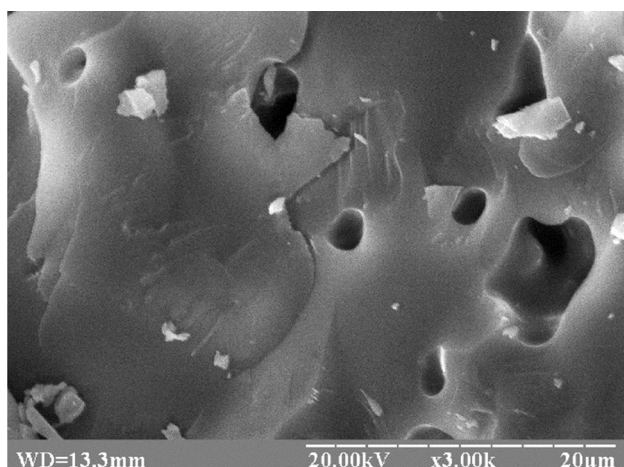


Fig. 6. Microstructure of ceramic material of composition No. 3, firing mode II

The high degree of sintering of quartz ceramics, which can be observed on the micrograph of the sample, is achieved due to the compaction of the structure. This occurs as a result of the uniform distribution of aluminum phosphate particles in the quartz glass, as confirmed by the absence of the “ $\text{SiO}_2 - \text{AlPO}_4$ ” phase interface.

6. Discussion of the results of the effect of aluminum phosphate on the properties of quartz ceramics

In the work, the samples were made by the slip casting method, which is justified by the possibility of obtaining products of complex shape and, as a consequence, expansion of application areas. Quartz ceramics are traditionally used as heat-insulating elements in thermal units, as pipes for feeding molten aluminum, molds for casting metals, etc. However, the use of quartz ceramics for the manufacture of fairings and various elements of rocket and space technology is the most effective. The operation of quartz ceramic products is characterized by extreme conditions, namely, sharp temperature drops, increased loads, aggressive medium, etc.

Already at the stage of manufacturing the semi-finished product, it was noted that the slip with AlPO_4 additives does not delaminate, is homogeneous and cast well into plaster molds. The semi-finished product is easily removed from plaster molds, which is a very significant factor, since quartz ceramic products usually have minimal shrinkage and difficulties in removing from molds.

The experiments revealed that the addition of aluminum phosphate in an amount of 10 wt % to quartz glass practically does not change the properties of the samples obtained (Table 1). The most preferred values of properties after firing are characteristic of samples of composition No. 3, in which the amount of AlPO_4 is 20 wt %. The ultimate compressive strength of such ceramics increases by almost 1.5 times compared to the original composition. The porosity in this case is ~5 %.

It should be noted that up to a temperature of 1,200 °C, no noticeable phase and modification transformations occur in the samples (Fig. 1), which should indicate the stability of the structure under study.

The study of the influence of firing conditions and temperature-time modes for ceramics of composition No. 3 shows that the samples fired in the sand backfill turned out to be quite brittle. Therefore, the ultimate compressive strength and CLTE could not be measured. A fairly significant increase in ultimate compressive strength was observed during the firing of the samples in the chrome oxide backfill ($\sigma_{\text{comp}}=135\text{--}136\text{ MPa}$) (Fig. 3). However, their CLTE values also significantly increased to $(40\text{--}90)\cdot 10^{-7}\text{ deg}^{-1}$, which is unacceptable and indirectly indicates a significant amount of cristobalite phase in the resulting ceramics.

It was found that the optimal firing temperature is 1,100–1,200 °C, the firing time is 1–2 h, and the firing medium is oxidizing. The samples obtained in this way are characterized by $\alpha=31.6\cdot 10^{-7}\text{ deg}^{-1}$; $\sigma_{\text{comp}}=153\text{ MPa}$; $P=2.7\%$;

$tg\delta$ (frequency 10^{10} Hz, temperature $20\text{ }^{\circ}\text{C}$) 0.001; ϵ (frequency 10^{10} Hz, temperature $20\text{ }^{\circ}\text{C}$) 10 (Fig. 2, 3).

It should be noted that the firing of the samples is carried out using a technology fundamentally different from the traditional one. Namely, the products are placed in a furnace at a maximum firing temperature ($1,100\text{ }^{\circ}\text{C}$), held for 2 hours, and removed from the furnace immediately after firing. Subsequent cooling of the samples is carried out in the air under natural conditions. Due to high temperature resistance, no defects occur on the products. Thermal resistance depends on the nature of the chemical, crystal structure, micro- and macrostructure. The thermal resistance of ceramics is greatly influenced by the size and shape of the product, the difference in the values of the operating temperatures and the rate of their change. Short firing makes it possible to avoid the formation of cristobalite (as confirmed by X-ray phase analysis), which usually significantly reduces the properties of quartz ceramics (Fig. 5). The high degree of sintering of quartz ceramics, which can be observed in the micrograph of the sample (Fig. 6), is achieved due to the compaction of the structure. This occurs as a result of the uniform distribution of aluminum phosphate particles in the quartz glass, as confirmed by the absence of the “ $\text{SiO}_2 - \text{AlPO}_4$ ” phase interface.

Short firing also contributes to significant power savings and increases production performance due to the possibility of firing several series of products per day and, accordingly, increasing productivity.

Thus, a method for manufacturing quartz ceramic products with reduced porosity, increased strength characteristics and simplified firing technology has been developed. This method can be used for making a wide range of products due to the high packing density of the material in liquid and solid states. High packing density is achieved by adding 10–20 wt % aluminum phosphate into the quartz glass slip.

7. Conclusions

1. The studies showed the effect of aluminum phosphate additives in the amount of 10–20 wt % on the properties of quartz ceramics. According to the totality of properties, the addition of AlPO_4 in an amount of 20 wt % has the most positive effect, which is noted already at the stage of preparation of the semi-finished product. The original slip is characterized by manufacturability, no pre-treatment of plaster molds is required, the semi-finished product is extracted from them without the difficulties associated with quartz ceramics. Up to a temperature of $1,200\text{ }^{\circ}\text{C}$, according to differential thermal analysis, no noticeable phase and modification transformations occur in the samples, which indicates the stability of the structure under study.

2. The influence of firing mode and conditions on the properties of quartz ceramics with the addition of 20 wt % aluminum phosphate was studied. The most preferable firing mode is as follows: oxidizing medium, the samples are immersed in a hot furnace, after being held at the maximum temperature, they are removed from the hot furnace; samples are cooled in the air. The materials obtained in this way are characterized by the following properties: $\alpha=31.6\cdot 10^{-7}\text{ deg}^{-1}$; $\sigma_{\text{comp}}=153\text{ MPa}$; $P=2.7\%$; $tg\delta=0.001$ (frequency 10^{10} Hz, temperature $20\text{ }^{\circ}\text{C}$); $\epsilon=10$ (frequency 10^{10} Hz, temperature $20\text{ }^{\circ}\text{C}$).

3. It is noted that the main crystal phase prevailing in the obtained ceramic materials is aluminum phosphate. The microstructure of the sample is characterized by the presence of a small number of residual pores and a densely sintered shard.

It was found that adding aluminum phosphate to quartz ceramics can significantly reduce the duration and temperature of firing products. This will significantly reduce power consumption, improve production performance and increase the basic properties of polyfunctional quartz ceramic products.

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